Indian Journal of Engineering & Materials Sciences
Vol. 11, June 2004, pp.224-226

Note

An electrolytic method for studying the microstructure and grain size of Al-1.25% Mn alloy

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Received 31 July 2003; accepted 5 April 2004

An electrolytic polishing and anodizing technique for studying the microstructure and grain structure of Al and Al-1.25% Mn alloy samples is described. The same electrolyte, based on phosphoric acid, can be used for both the applications under slightly different experimental conditions.

IPC Code: Int. Cl.[7] C 25 C 3/06

Determination and control of grain size in wrought aluminium and its alloys are important, especially for sheets subjected to extensive cold forming operations. For deep-drawing applications, the sheets should have a fine grain size together with a balanced texture. Grain refinement in Al and its alloys, is achieved by cold working and annealing treatments. Direct chill cast Al alloys consist of supersaturated solid solutions. During homogenization treatment, the alloying elements will be largely precipitated. The presence of the alloying elements either in solid solution or as precipitates will affect the recrystallization characteristics and hence the grain size[1,2]. During the investigations[1] on precipitation and recrystallization in an Al-1.25wt.% Mn alloy, conventional metallographic techniques were found to be inadequate to reveal the fine precipitates as well as the grain structure. Therefore, a modified electro-polishing and anodizing technique was developed for preparing the specimens for optical microscopy.

Electro-polishing involves the anodic dissolution of a metal under suitable conditions and results in a smooth and brilliant surface. Since no mechanical action is involved, the polished surface will be free of scratches, strains and embedded abrasives[3]. Specimen preparation by electro-polishing is very attractive because it is simple and quick, especially when employed for rolled plate and sheet specimens which require little surface preparation. Generally, electrolytes based on perchloric acid are recommended[3-5] for use with commercial Al and its alloys.

Conventionally polished metallographic specimens have to be etched, i.e., treated with dilute aqueous or alcoholic solution of acids, in order to remove the deformed surface layers and to reveal the microstructure. In a polycrystalline specimen due to differences in the reactions rates at the grin boundaries and in the bulk of the grains the latter will be clearly delineated. This will enable determination of the grain size of the polished and etched specimen. However, in the case of commercial Al and some of the alloys like those containing Mn, the conventional etchants do not satisfactorily reveal the grain structure. Hence, the polished specimens are generally subjected to anodizing treatment in a fluroboric acid electrolyte[3]. During anodizing, an oxide film is formed on the surface of the polished specimen which is examined under polarized light. The mechanism of contrast has been described by Gifkins[6]. The oxide film formed on the surface of the specimen develops furrows which are characteristic of the underlying grains. When plane polarized light is incident on such an oxide film, it undergoes double reflection which introduces some ellipticity. The analyzer cannot extinguish such light completely and some of it will be transmitted. This gives rise to bright and dark contrast from grains having different orientations. This technique also enables the detection of preferred orientation since grains of same orientation will appear alike. In this note, the composition and operating conditions of a modified electrolyte based on ortho-phosphoric acid for both polishing and anodizing of Al-1.25 wt.%Mn alloy specimens are given.

Experimental Procedure

The composition and operating conditions of the electrolyte are given in Table 1. The electrolyte was prepared by mixing analytical grade ortho-phosphoric acid, ethylene glycol, hydrofluoric acid and distilled water in the proportions indicated, at room temperature. For experimental purposes, about 200 mL of the electrolyte was taken in a glass beaker. For all experiments, rectangular specimens of 2×1 cm^2 were

Table 1 — Composition of the electrolyte and the operating conditions.

Composition (vol %)			Operating conditions	
			Polishing	Anodizing
Orthophosphoric acid	60 - 65	d.c. Volt.	30 - 50	40 - 60
Ethylene glycol	30 - 35	Temp.(˚C)	50 - 70	15 - 25
Hydrofluoric acid	2 - 3	Time (min)	5-20	3-5
Distilled water	3 - 5			

used. Annealed sheet specimens were generally 0.1-0.2 cm thick and specimens of up to 0.5 cm in thickness have been used. The specimen was made the anode and a flat piece of graphite of 2.5×1.5×0.3 cm^3 was used as cathode. The experimental arrangement was similar to that described by Thomas[7]. The same electrolyte could be used for both polishing and anodizing but with different operating conditions. Good polishing was obtained with the electrolyte maintained at 50-60 °C using a d.c. voltage of 30-50 V. Polishing time has been found to vary, depending on the initial surface condition of the specimen, from 5 min to 20 min. Slow stirring of the electrolyte has been found to be helpful for maintaining the temperature and for rapid and uniform polishing. At the end of polishing, when the specimen develops brilliant silvery lustrous appearance, it is quickly washed in warm water, followed by ethanol and dried. The specimen can now be used for optical microscopy. Fig. 1 shows the microstructure of direct chill (d.c.) cast and homogenized Al-1.25% Mn alloy. The fine size and non-uniform distribution of the disc shaped α-Al-Fe-Mn-Si precipitates may be noted.

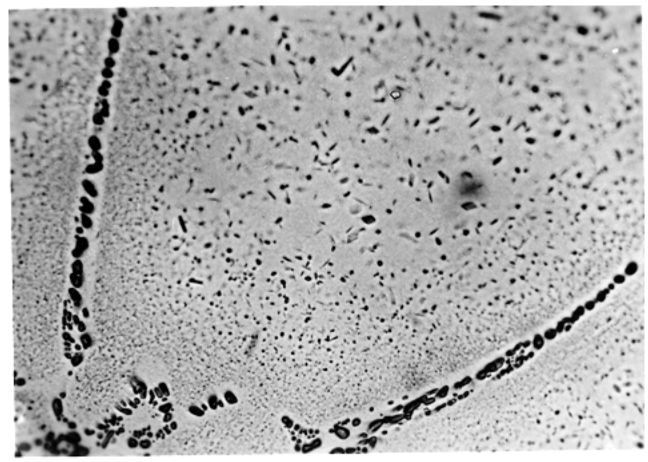

Fig. 1 — Microstructure of d.c. cast and homogenized Al-1.25% Mn alloy showing the variation in the size and distribution of α-Al-Fe-Mn-Si precipitates. Specimen electropolished after progressive grinding up to 6/0 grit SiC paper (*20 μm*)

Results and Discussion

In order to reveal the grain structure by anodizing the electrolyte was cooled and maintained at 15-25 °C On applying a d.c. potential of 40-60 V, the current initially rises and then drops to a very low value of 0.02-0.04 A. Anodizing time of 3-5 min has been found adequate. The photomicrographs in Fig. 2, recorded under polarized light, are from annealed specimens of Al-1.25% Mn alloy consisting of (a) fine equi-axed grains and (b) somewhat coarser grains with some preferred orientation.

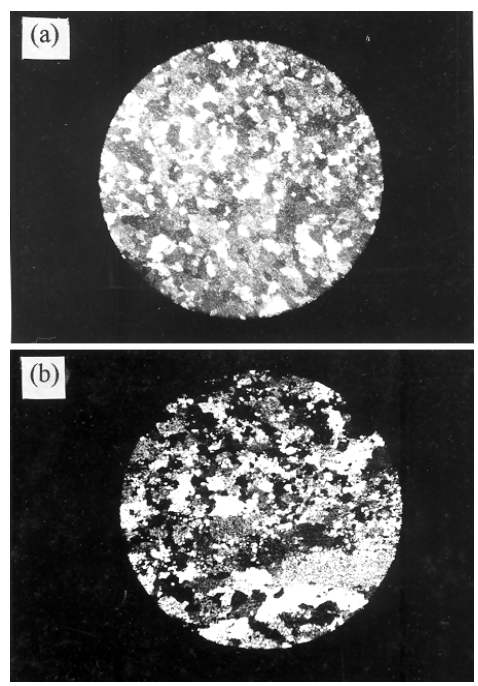

Fig. 2 — Grain size of cold rolled and annealed sheet specimens of Al-1.25% Mn alloy showing: (a) fine grain size and (b) non-uniform grain size with some preferred orientation. Specimens electropolished and anodized without any surface preparation. (Polarized light) (*100 μm*)

It has been found that when sheet specimens with flat edges are used, polishing and anodizing take place simultaneously on all surfaces. This enables the study of the microstructure and grain size along the long and short transverse directions of the specimen. Fig. 3 shows the grain structure of an annealed specimen along the surface and the two transverse directions. Use of a tint plate imparts colour contrast to the

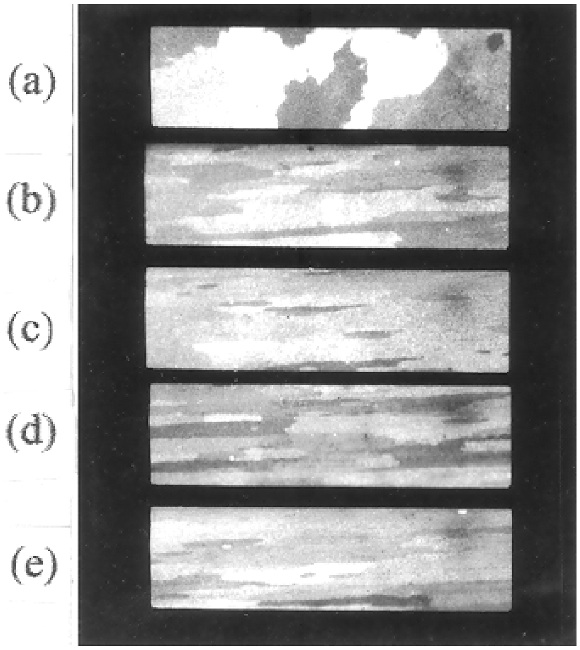

Fig. 3 — Grain structure of annealed Al-1.25% Mn alloy sheet along: (a) surface, (b&c) Long transverse direction, (d & e) Short transverse direction. (b-e) from different locations of the same specimen. Polarized light (*100 μm*)

grains, which besides being pleasing to the eyes is quite helpful during grain size measurement. On account of the short polishing times, the electrolytic method is also used for polishing specimens used for Brinell and Vicker's hardness testing. The high reflectivity of the specimen facilitates easy measurement of the size of the indentation.

Conclusions

The experimental electrolyte has been successfully used to study the microstructure and grain size of commercial aluminium and Al-1.25% Mn alloy. The present technique has been found to offer the following benefits: (i) Rapid and uniform polishing and anodizing of Al and Al-1.25% Mn alloy specimens; (ii) Simultaneous polishing of surfaces and edges of the specimen; (iii) It be used for rapid polishing of specimens for hardness testing; and, (iv) Even very thin and small specimens can be rapidly polished without the necessity of fixing in plastic mounts.

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